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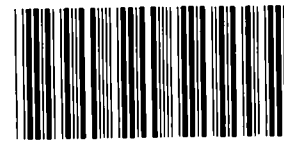
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Baseline Nox Emissions during Combustion of Wood-Derived Pyrolysis Oils

Presented at the Specialists *Workshop on Biomass Pyrolysis Oil Combustion*, Estes Park, CO, 1994.

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Baseline NO_x Emissions During Combustion of Wood-Derived Pyrolysis Oils

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ABSTRACT

NO_x emissions from two pyrolysis oils of similar origin and overall composition but differing nitrogen contents (0.12 and 0.32 % of dry fuel) are determined in a pilot-scale combustor. No NO_x reduction technology is employed in these tests, establishing the baseline or uncontrolled levels of NO_x. Measured effluent oxygen concentrations range from near 0% to near 21%, with stoichiometric ratios ranging from 0 to 1. NO and NO_x are measured separately and found to differ by insignificant (≈ 10 -25 ppmv) amounts. Other relevant gas species (CO₂, CO, total hydrocarbons, and O₂) are also reported.

Peak NO_x emissions from these fuels vary from about 300 to around 650 ppmv, with lower levels associated with low nitrogen content fuels. Trends with stoichiometric ratio and fuel nitrogen content agree qualitatively with behavior from other nitrogen containing fuels, including biomass, coal, and petroleum oils. Nitrogen conversion efficiencies as a function of stoichiometric and fuel nitrogen content are observed to decrease with increasing fuel nitrogen content and increase with increasing oxygen content. Measurements of thermal, prompt, and fuel NO_x contributions indicate that fuel NO_x is the dominant formation mechanism for these fuels. These data suggest that NO_x formed during combustion of pyrolysis oil lends itself to many of the same control technologies as are used in other nitrogen-containing fuel.

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INTRODUCTION

The combustion behavior of biomass-derived pyrolysis oils under conditions similar to those found in commercial boilers is largely uncharacterized. One of the major issues associated with such combustion is the production of pollutants. Of primary interest is the production of NO_x . Since such oils are ultimately targeted toward both boiler and turbine applications, NO_x production under a wide range of stoichiometries is of concern.

The nitrogen-containing, combustion-generated compounds of greatest concern are nitric oxide (NO), nitrogen dioxide (NO_2), and nitrous oxide (N_2O). The term NO_x generally refers to the former two compounds or, when used more broadly, to all three. NO is the most studied and, in most cases, the most significant of the three compounds. Equilibrium NO concentrations depend on both temperature and stoichiometry. At flame temperatures (2000 K), equilibrium values of NO range from about 3000 ppmv at an equivalence ratio of 0.8 to about 30 ppmv at an equivalence ratio of 1.2 [Bowman, 1991]. At exhaust temperatures (300 K), equilibrium concentrations of NO are uniformly less than 10 ppmv. Exhaust NO emissions from combustion systems are rarely as high as their equilibrium value at the flame temperature, but are generally higher than their equilibrium value at the exhaust temperature, indicating that both NO formation and destruction are influenced by chemical kinetics.

Formation mechanisms for NO_x are generally divided into three categories: thermal NO_x , fuel NO_x , and prompt NO_x . Each is discussed here in terms of the NO, which is typically the most significant component of NO_x . Thermal NO has the most completely understood mechanism from a chemical kinetic viewpoint. It depends on the relatively slow (slow compared to most combustion reactions) conversion of molecular or atomic nitrogen to NO by oxygen and hydroxyl radicals. The formation of thermal NO is strongly dependent on temperature, moderately dependent on oxygen concentration, and nearly independent of pressure.

Nitrogen-containing fuels produce nitrogen-containing radicals in flames that can greatly increase the amount of NO observed in the vitiated flow by the fuel NO_x mechanism. Fuel-bound nitrogen is generally considered to generate some gas-phase intermediate species (HCN or some derivative is most commonly cited) that competitively reacts to form either molecular nitrogen or NO. The details of the kinetics are less well understood than those of thermal NO formation, but parametric studies indicate general trends. NO levels increase with increasing fuel nitrogen contents, but the dependence is less than first order. Therefore, the *fractional* conversion of fuel nitrogen to NO generally decreases as the amount of fuel nitrogen increases even though the *total* amount of NO increases with increasing fuel nitrogen [Pohl and Sarofim, 1976]. Most studies conclude that the form of nitrogen in the parent fuel has little impact on NO concentrations [Bowman, 1992], although most of the work appears to have been performed on aromatic compounds not containing amine functional groups. Biomass-bound nitrogen is believed to occur in amine form to a much greater extent than in other fuels (notably, petroleum-derived oils and coal). If pyrolysis conditions are sufficiently moderate to prevent conversion of amine nitrogen to other forms, the fate of the amine nitrogen during combustion may be quite different in the biomass-derived pyrolysis oils than that of nitrogen from other fuels.

Prompt NO is the final mechanism for NO formation. Prompt NO refers to near-flame concentrations of NO that are higher than can be explained by the thermal mechanism alone. Proposed mechanisms for prompt NO formation involve both hydrocarbon radical intermediates and oxygen or hydroxyl radicals [Bowman, 1991]. The mechanism involving hydrocarbon intermediates has many similarities with that of fuel NO.

Other forms of NO_x (NO₂, N₂O) contribute to the total emission. NO₂ is formed at relatively low temperatures, and is rapidly reconverted to NO at high temperatures (lifetime of ≈ 10 ms @ 1500 K). N₂O is also a low-temperature species, with lifetimes shorter than those of NO₂ at higher temperatures. Most recent measurements indicate that NO₂ is a minor component of conventional, high-temperature combustion systems [Bowman, 1992].

The critical engineering issues in NO_x formation from pyrolysis oils are the overall NO_x emissions and their sensitivity to combustion of specific types of pyrolysis oils as well as operating conditions. In most of the results reported below, total NO_x, rather than NO, is reported. In these cases, the measurements are performed by passing the sampled effluent from the MFC through a catalytic device that reduces NO₂ to NO. The concentration of NO is measured after this conversion. Bypassing the converter renders an NO, as opposed to NO_x, measurements. We occasionally bypass the converter to record the difference between NO and NO_x measurements. In these data, the difference amounted to up to 30 ppmv. This difference is negligible for the purposes of our discussion below. The sampling system has been described in earlier reports. Calibration gases are introduced in this system at the probe tip, not the analyzers, to verify that reactions on sampling system surfaces do not influence the results.

EXPERIMENTAL APPROACH

Concentrations of NO, NO_x (as NO₂), SO₂, CO, total hydrocarbons (THC as CH₄), CO₂, and O₂ were monitored by continuous analyzers sampling the exit gas from Sandia's Multifuel Combustor (MFC). This laboratory unit is designed to simulate commercial-scale combustion furnaces, but is highly instrumented with diagnostics and controllers to provide detailed characterization of gas compositions and reactions, solids reactions, and surface interactions. The combustor, depicted in Figure 1, is a 4.2 m high vertical turbulent-flow furnace with a 15 cm inner diameter. The upper six of seven furnace modules are electrically heated and independently controlled. The furnace duct is open at the bottom, and discharges across a 0.7 m high open space into the inlet of a large duct, which also draws laboratory air for dilution and cooling prior to exhaust. For the purposes of emissions monitoring, a sampling probe is inserted into the undiluted and uncooled exhaust gas through the lowest furnace section below the fuel injection point. A natural gas burner situated at the top of the furnace can supply a preheated, vitiated oxidant flow to aid in simulating full-scale furnace conditions or to compare gas compositions from non-nitrogen containing fuels (methane or natural gas) with those from nitrogen-bearing fuels. NO_x emissions in the electrically heated flow, without combustion of pyrolysis oil or natural gas, were below 10 ppmv with the furnace wall and exit gas temperatures at 900°C. Under the same operating conditions, but with a natural gas flame, NO_x emissions were between

30 and 40 ppmv. By comparison, peak NO_x concentrations for the pyrolysis oils tested here ranged from 250 to 650 ppmv.

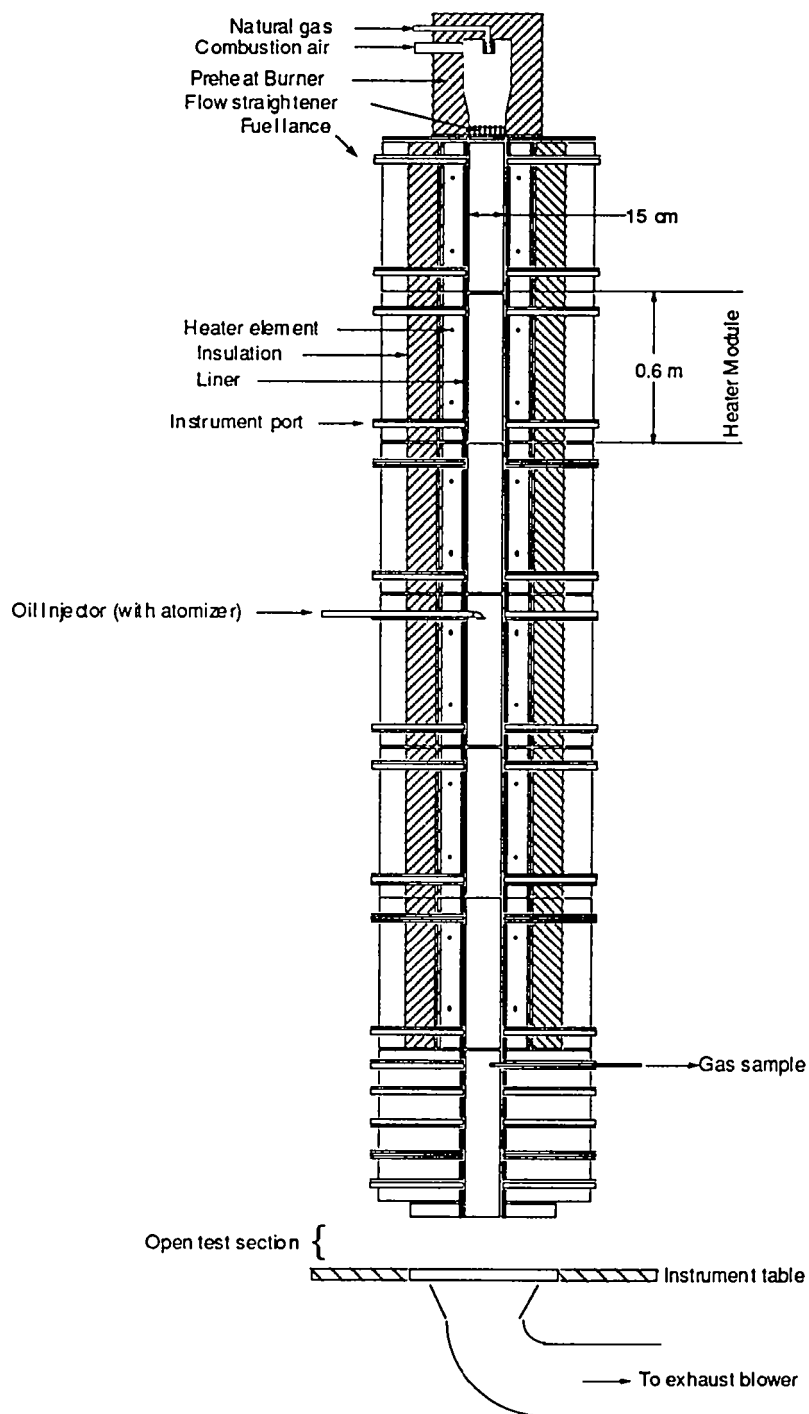


Figure 1 Schematic diagram of Sandia's Multifuel Combustor as used in these studies of pyrolysis oils.

CO₂, CO, NO, NO_x, and SO₂ concentrations are measured using nondispersive infrared (NDIR) analyzers, O₂ concentrations by a paramagnetic analyzer, and total hydrocarbons (THC) by a flame ionization detector. The maximum CO concentration that could be measured was 1,000 ppmv, which was exceeded in many cases when approaching an equivalence ratio of unity. All concentrations were measured on a dry gas basis. The instrumentation is calibrated before and usually after each run by introducing calibration gases at the sample probe tip. No interferences between measured species are evident in either the calibration or the instrument characterization tests. All gas concentrations were measured on a dry-gas basis, with water removed by refrigeration. Sample lines are heat traced up to the refrigeration point to avoid condensation on the teflon sample train walls.

FUEL CHARACTERISTICS

The oil used to generate all of the data reported below was produced by Ensyn Technologies Inc., Greely, Ontario, Canada and were provided to Sandia by NREL. A dry, hardwood feed stock was used to produce the oil. The properties of this pyrolysis oil are clearly distinguished from those of traditional fuel oils, such as petroleum distillates. The presence of significant water in the liquid pyrolysis products disqualifies them as oils by some strict definitions [Lapedes, 1978]. They occasionally exhibit strong acidic tendencies and often contain some suspended solids, neither of which properties are common in traditional oils. Although reference to these fuels is made here and elsewhere as pyrolysis oils, these chemical differences play significant roles in their handling and some combustion properties.

Table 1 indicates the relevant properties of these pyrolysis oils. Compared with traditional petroleum-derived oils, the pyrolysis oils contain much more moisture, oxygen, and ash, comparable amounts of nitrogen, sulfur, and hydrogen (dry basis), and have about half of the heating value.

**Table 1 Properties of the Ensyn Pyrolysis Oils Relevant to
NO_x Emission Studies (dry basis)**

	Oil #1	Oil #2
Moisture	25	27
C	59.23	58.64
H	10.25	10.07
O	29.10	29.21
N	0.32	0.12
S	0.05	0.01
Ash	1.05	1.05
Heating Value, daf	23.1	23.0
MJ/kg (BTU/lb)	(9888)	(9919)

RESULTS

The focus of our work and the bulk of the discussion is on NO_x generation. A brief report of other major gas species is included after the discussion of NO_x .

NO_x Emissions

Combustion tests of the low-nitrogen content oil (Ensyn pyrolysis oil #2) produced the data that are reported in Fig. 2 on both an as-measured and corrected basis. As the overall stoichiometry changes, the concentration of NO_x varies in part due to changing combustion temperatures and kinetics and in part due to the dilution of the vitiated flow by changing ratios of fuel to air. The data are corrected to remove the latter effect by normalizing the measured value of NO_x . The normalization results in the concentration of NO_x that would have been produced had the combustor been fired at some arbitrary excess oxygen level. There is no general agreement on what oxygen concentration should be chosen for this normalization. We report the data on a 3% O_2 basis – the same basis on which many pollutant emission regulations are based.

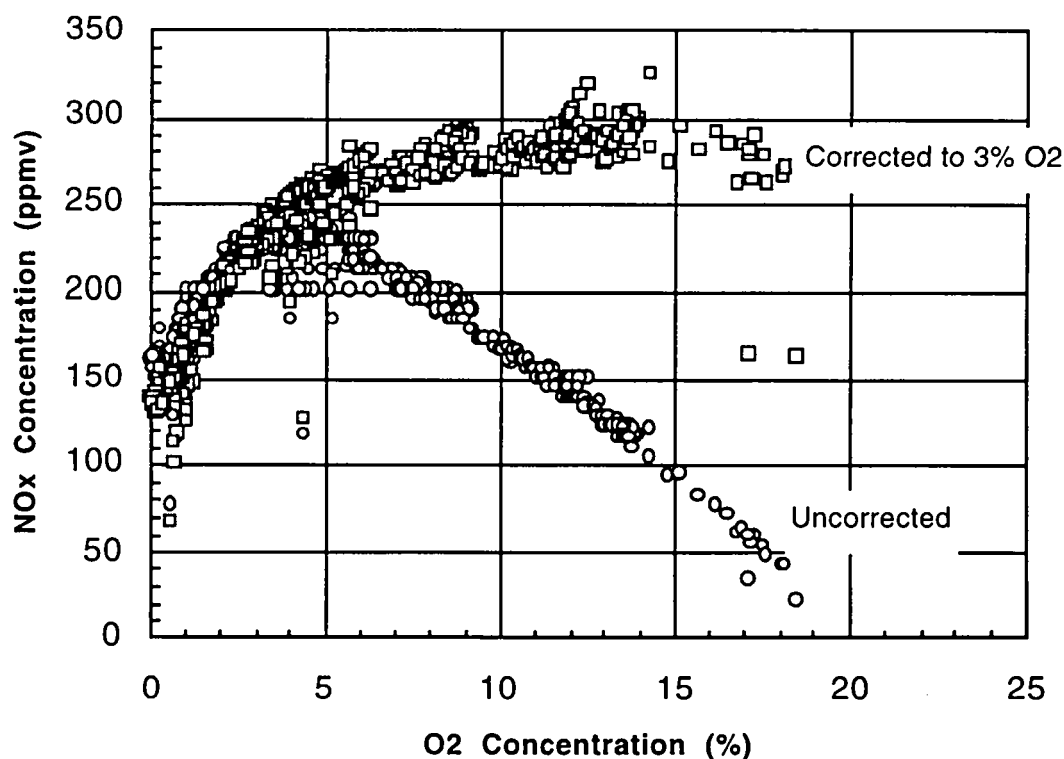


Figure 2 Measured NO_x emissions from the Ensyn 1 pyrolysis oil fired in the MFC as a function of measured effluent oxygen concentrations.

Another common basis for reporting such data is the calculated equivalence ratio based on complete combustion of the reactants. As the equivalence ratio approaches unity, the calculated amount of O_2 in the vitiated flow assuming complete combustion may differ significantly from the actual value due to the formation of CO or other partially oxidized species.

The data indicate a rapid increase in NO_x with increasing oxygen concentration in the oxygen mole fraction range of 0 to about 2 %. At oxygen concentrations greater than about 2%, the raw data indicate a monotonic decrease in NO_x emissions with increasing oxygen concentration. This trend is due almost entirely to the dilution of NO_x by increased combustion air. When corrected for the dilution effect, the NO_x levels are seen to stabilize, with essentially no change with increasing oxygen concentration. Both of these observations are consistent with the combustion of other nitrogen-containing fuels. The initial increase represents the increased formation of NO_x with increasing availability of oxygen in the flame zone. At some point, the formation of NO_x becomes independent of oxygen concentration (large excess oxygen), therefore the only changes in NO_x concentration noted with increasing O_2 are the dilution effect.

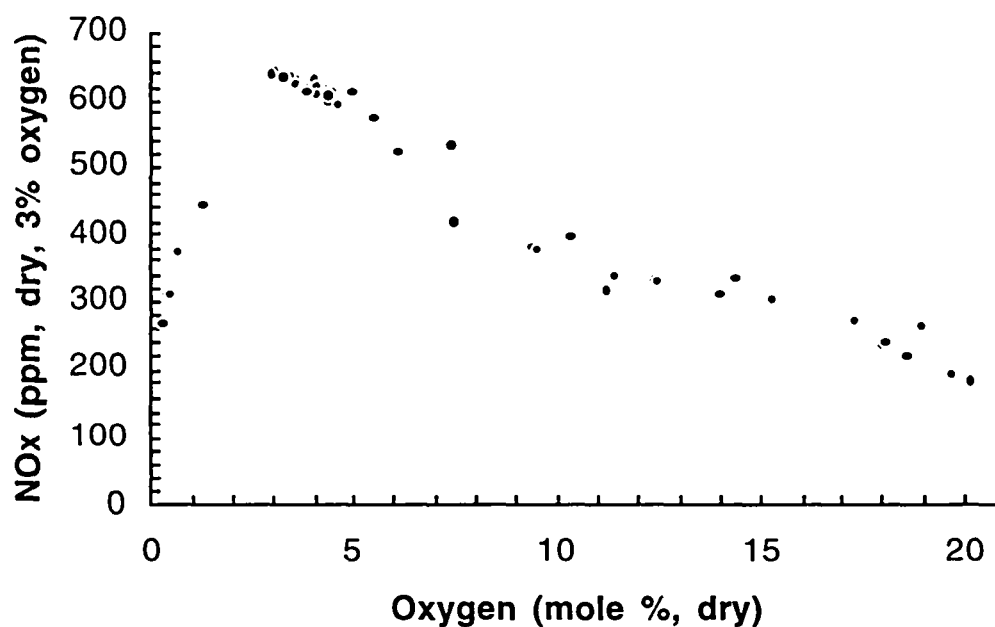


Figure 3. NO_x concentrations (normalized to 3% O_2) from combustion of the high-nitrogen content pyrolysis oil in the MFC.

Data from the high-nitrogen content fuel (Oil #1) are illustrated in Fig. 3 and do not exhibit the plateau in NO_x concentrations at high oxygen concentrations seen in Fig. 2. In the

experiments with Oil #1, flame temperatures changed significantly as we changed oxygen concentrations because the atomizing air ratio was changed significantly. In the experiments with Oil #2, effluent oxygen concentration was changed by changing the secondary air, which has little influence on the temperature and stoichiometry of the flame. Therefore, flame temperature did not change significantly.

Other qualitative results are also consistent with data from combustion of other nitrogen-containing fuels. For example, NO_x concentrations were also measured in the MFC at the same gas temperature ($\approx 1000^\circ\text{C}$) but with no combustion of pyrolysis oil or any other fuel (temperature rise was achieved through electrical heating). NO_x levels under these conditions were uniformly less than 10 ppmv. NO_x concentrations measured during the firing of natural gas at the same overall temperature were uniformly less than 40 ppmv. The increase relative to electrical heating is presumed to be associated with the generation of radicals in the flame, which increases the generation of NO_x in the combustion zone. These results suggest that thermal NO_x makes only insignificant contributions to the overall NO_x concentrations observed in the gases. This, too, is consistent with results from other nitrogen-containing fuels.

Similarly, the quantitative results do not differ greatly from data on the combustion of other fuels. For example, uncontrolled NO_x emissions from coal- and oil-fired power plants operating with about 3% oxygen in the flue gas and with nitrogen concentrations in the fuel of about 0.4 % result in NO_x emissions of approximately 700 ppmv. However, peak temperatures in both coal and petroleum-oil flames exceed those in the pyrolysis oil flames by several hundred Kelvins. Since NO_x emissions increase with increasing temperature, these results suggest that pyrolysis oils tend to form slightly more NO_x than other similar fuels, all else being equal.

A major difference between the results from Ensyn Oil #2 and those from Ensyn Oil #1 is the peak NO_x concentration. This is related to the amount of fuel nitrogen in the oils. As indicated in Table 1, the fuel nitrogen level in Ensyn Oil 1 is about 2.7 times greater than that of Ensyn Oil 2. The observed increase in peak effluent NO_x concentration is consistent with this change in fuel nitrogen. However, the NO_x concentrations do not increase in proportion to fuel nitrogen content. The peak NO_x concentration measured in our combustion tests increases by a factor of about 2, whereas fuel nitrogen content increases by a factor of 2.7. This, too, is consistent with trends from other nitrogen-bearing fuels.

Other Major Gas Species

Changes in other major gas-phase species were also recorded during these tests. Figure 4 illustrates the CO concentration as the combustor stoichiometry approaches an equivalence ratio of unity. CO concentration increases from negligible to about 1%, with the highest measured values occurring the lowest measured oxygen concentrations (about 0.7 % oxygen). These conditions correspond essentially to an equivalence ratio of 1 (i.e., all of the excess oxygen would be consumed if the CO were to convert to CO_2). The abscissa in Figs. 2 through 4 represents measured O_2 concentrations, not calculated values based on flow rates.

Increases in CO concentrations correspond closely to the observed decreases in NO_x concentrations. Both reflect the competition between species for oxygen as the available oxygen becomes limited. The conversion of carbon to CO is relatively rapid and decreases the amount of oxygen available for NO_x formation. However, the conversion of CO to CO₂ is relatively slow and, in the presence of a limited oxygen supply, CO concentrations are appreciable.

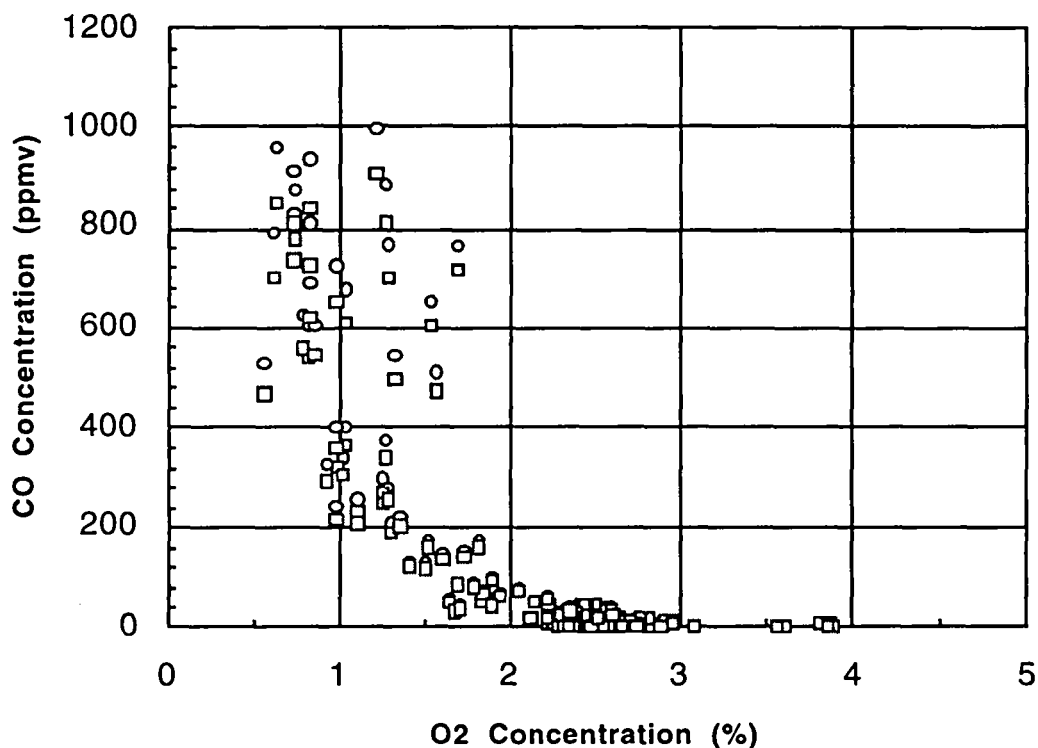


Figure 4 CO concentration as a function of measured oxygen concentration in the MFC during combustion of a pyrolysis oil. Squares represent corrected values. Circles represent uncorrected vlaues. Correction to 3% oxygen makes little difference over the limited range of oxygen concentrations with measureable CO concentrations. Compare with Figs. 2 and 5.

Figure 5 illustrates the trend in observed CO₂ concentrations with changing oxygen concentrations. The only significant trend is the effect of correcting the data to 3 % O₂. There should be small decreases in the CO₂ concentrations associated with the changes in CO concentration illustrated in Fig. 4, but the changes in CO₂ are insignificant ($\approx 1\%$) compared to the overall CO₂ concentration (16 %).

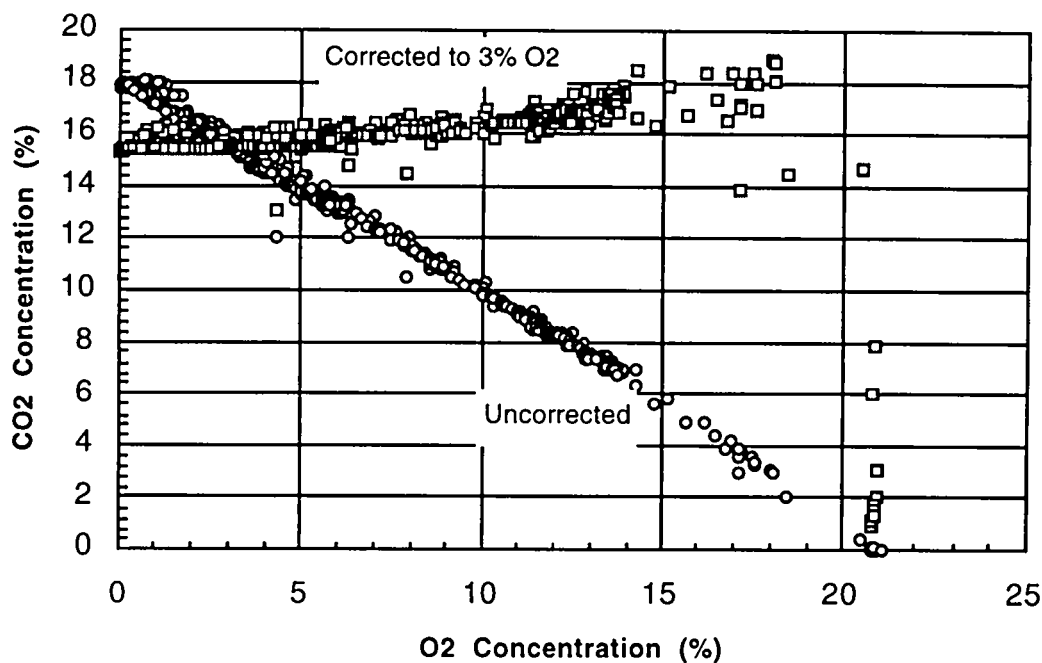


Figure 5 CO₂ concentration as a function of measured oxygen concentration in the MFC during combustion of Ensyn pyrolysis oil #2. Compare with Figs. 2 and 3.

These results suggest that NO_x controls used for other nitrogen-containing fuels will work with equal success with pyrolysis oils. NO_x emissions should be controllable using staged combustion, low-NO_x burners, or other similar approaches.

In combustion systems such as this, where overall NO_x emissions are dominated by fuel nitrogen, the fuel nitrogen conversion efficiency is a convenient term to use in discussing trends. The conversion efficiency is the ratio of NO_x molecules in the effluent to fuel nitrogen atoms in the feed. The normalization to 3% O₂ is not required to determine this ratio. The fuel nitrogen conversion efficiency can exceed 100% if there is significant contribution to NO_x by thermal or some prompt mechanisms. In practice, conversion efficiencies for fuels containing significant nitrogen are in the range of 20-80 %.

CONCLUSIONS

NO_x measurements were completed for two pyrolysis oils produced by the same process but differing in fuel nitrogen content. Combustion tests were conducted in a

These data indicate: (1) fuel nitrogen conversion efficiency increases with increasing oxygen concentration if flame temperature is held constant, (2) fuel nitrogen conversion efficiencies decrease with increasing fuel nitrogen content (NO_x levels increase, but conversion efficiencies decrease), and (3) NO_x emissions are dominated by NO, which is primarily formed from fuel nitrogen.

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